


PILLSBURY WINTHROP

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1000 TYSONS BOULEVARD MCLEAN, VA 22102 703.905.2000 F: 703.905.2500

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[0020]

[Test 1] Phosphoric acid adsorption test

(1) Preparation of iron hydroxide

1M NaOH aqueous solution was added dropwise to 1M FeCl₃ aqueous solution to obtain a gel precipitate, pH7. The said iron hydroxide gel precipitate was passed under suction through No. 1 filter paper and washed with distilled water. After the said washing had rendered the filtrate sulphuric acidic, silver nitrate solution was added until a white suspension was no longer produced. In this, about 10 l of distilled water was used per 250ml of starting iron chloride solution. After the said washing, the precipitate was dried at 50°C for approximately 24 hours (as the precipitate formed into hard pellets after about 10 hours of drying, this was powdered with a pestle and then the drying process was repeated). The powder thus obtained had an Fe content of 50.2%. This was stored on silica gel in a desiccator with reduced interior pressure.

[0021] (2) Comparison Preparation A1

Commercially available dehydrated aluminium hydroxide gel (Japanese Pharmacopoeia) was used for purposes of comparison. This had an Al content of 52.8%.

[0022] (3) Test methods

500 ml of a 10 mM KH₂PO₄ test solution at 37°C was pH adjusted with hydrochloric acid or sodium hydroxide. 1g of either the said iron hydroxide (1) or dehydrated aluminium hydroxide gel (2) was added to the said test solution, mixed, sampled over time and then filtered through a membrane filter with 0.45µm pore size. The phosphoric acid content of the filtrate was measured by the phosphor molybdenum blue method. The concentrations of Fe and Al were measured using the frameless atomic absorption method.

[0023] (4) Results

The phosphoric acid adsorption of iron hydroxide and dehydrated aluminium hydroxide gel and the phosphorus adsorption of iron hydroxide and dehydrated aluminium hydroxide gel after 3 hours at different pHs are shown in Figure 3.

[0024] The vertical axis of Figure 1 is the quantity of phosphoric acid adsorbed to iron hydroxide (PBC/mmol and PBC/mg) and the horizontal axis is the time after addition to the test solution (t/minutes). In Figure 2, the vertical axis is the quantity of phosphoric acid adsorbed to dehydrated aluminium hydroxide gel (PBC/mmol and PBC/mg) and the

horizontal axis is the time after addition of the test solution (t/minutes). In each of these graphs, values are plotted for the different pH values. In Figure 3, the vertical axis is the quantity of phosphoric acid (PBC/mmol and PBC/mg) adsorbed to iron hydroxide (shown as Fe) and dehydrated aluminium hydroxide gel (shown as Al) and the horizontal axis is pH.

[0025] From these figures, it is clear that, at pH 2-8, the quantity of phosphoric acid adsorbed to iron hydroxide is greater than the quantity of phosphoric acid adsorbed to dehydrated aluminium hydroxide gel and there is a particularly large gap under more strongly acidic conditions.

[0026] In the said tests, the molar ratio between the phosphoric acid adsorbed and the quantity of Fe or Al added ($\text{PO}_4^{3-}/(\text{Fe}, \text{Al})$) was plotted at the different pHs and these plots, viewed as the effectiveness of phosphoric acid adsorption, are shown in Figure 4.

[27] From this figure, it is clear that Fe has a higher effectiveness for phosphoric acid adsorption than Al at all pHs and is markedly superior under more acidic conditions.

[28] The question of whether phosphoric acid adsorption by iron hydroxide is irreversible by changes in pH was also examined by a test in which phosphoric acid was adsorbed to iron hydroxide at pH 2 for 60 minutes by a method similar to that described above, and then the pH raised by 1 every 20 minutes subsequently and the adsorbed quantity measured.

[0029] The results obtained are shown in Figure 5.

[0030] From this figure, it is clear that the quantity of the phosphoric acid adsorbed at pH 2 that is subsequently lost when the pH is raised is less than predicted from the pH characteristics of the quantity adsorbed and thus that the phosphoric acid adsorption capability of iron hydroxide is irreversible despite an increase in pH.

[0031] Finally a comparison was made between the dissolution characteristics of Fe from iron hydroxide at pH 2, at which dissolubility is maximum, and the dissolution characteristics of Al from dehydrated aluminium hydroxide gel.

[0032] The results are shown in Figure 6 (where the vertical axis is the quantity of Fe or Al eluted out from iron hydroxide or dehydrated aluminium hydroxide gel (Fe or Al/mmol); and the horizontal axis is the time after addition to the test solution (t/minutes)).

[0033] It is clear from Figure 6 that almost no Fe dissolved in iron hydroxide whereas approximately 10% of Al was eluted out of dehydrated aluminium hydroxide.